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Nano Lett., Just Accepted Manuscript • DOI: 10.1021/acs.nanolett.7b03449 • Publication Date (Web): 23 Oct 2017

Downloaded from http://pubs.acs.org on October 23, 2017

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Capillary-force-assisted clean-stamp transfer of two-dimensional materials

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Keywords: 2D materials, stamp transfer, capillary force, heterostructure

Abstract:

A simple and clean method to transfer two-dimensional (2D) materials plays a critical role in the fabrication of 2D electronics, in particular, the heterostructure devices based on the artificial vertical stacking of various 2D crystals. Currently, clean transfer techniques rely on sacrificial layers or bulky crystal flakes (e.g., hexagonal boron nitride) to pick-up the 2D materials. Here, we develop a capillary-force-assisted clean-stamp technique that uses a thin layer of evaporative liquid (e.g., water) as an instant glue to increase the adhesion energy between 2D crystals and polydimethylsiloxane (PDMS) for the pick-up step. After the liquid evaporates, the adhesion energy decreases, and the 2D crystal can be released. The thin liquid layer is condensed to the PDMS surface from its vapor phase, which ensures the low contamination level on the 2D materials and largely remains their chemical and electrical properties. Using this method, we
prepared graphene-based transistors with low charge-neutral concentration \((3\times10^{10}\ \text{cm}^{-2})\) and high carrier mobility (up to \(48,820\ \text{cm}^2\ \text{V}^{-1}\ \text{s}^{-1}\) at room temperature), and heterostructure optoelectronics with high operation speed. Finally, a capillary force model is developed to explain the experiment.

Two-dimensional (2D) materials, such as graphene and transition metal dichalcogenides (TMDC), have stimulated extensive research efforts in the fields of nano-electronics and optoelectronics due to their exceptional electrical and optical properties \(^1,2\). By stacking different 2D material layers in van der Waals (vdW) heterostructures, new device concepts with outstanding functionalities have been demonstrated \(^3-6\). Both the full exploration of 2D material properties and the fabrication of heterostructures require a clean and intact transfer technique, to move these layered materials from the preparation substrates onto target positions without compromising their properties or qualities, and in particular, without any contamination. So far, polymethyl methacrylate (PMMA) has been widely used as the holder for transferring, owing to the simple yet versatile procedure\(^7\). However, PMMA always leaves a thin layer of long-chain molecules on the 2D material surfaces, even after exhaustive rinse with various organic solvents \(^8\). This residual contaminates the sample surfaces and has become a major limiting factor restricting the device performance. To solve this problem, sacrificial layers, such as a thin metal\(^9\),
or polylactic acid (PLLA) film\(^1\), have been used to separate PMMA from the 2D materials. However, it introduces extra fabrication steps including deposition, dissolving, and washing, which are tedious in particular in the preparation of heterostructures. Polydimethylsiloxane (PDMS)-based stamp transfer method has been exploited as a dry-transfer technique for nano-electronics \(^12,\ 13\). So far, it cannot peel off 2D monolayers directly from silicon dioxide (SiO\(_2\)) substrate because the adhesion energy between PDMS and 2D monolayers (~7 mJ/m\(^2\) for graphene)\(^14\) is far weaker than the vdW energy between 2D materials and SiO\(_2\) (~240 mJ/m\(^2\) for graphene)\(^15\). To increase the bonding energy between the dry-transfer stamp and 2D monolayers, atomically flat surfaces of hexagonal boron nitride (h-BN) flakes have been recently introduced \(^16-18\). As a trade-off, removing this BN layer to expose the functional 2D materials is challenging.

In this paper, we report a simple and universal capillary-force-assisted clean-stamp method to transfer 2D materials. We demonstrate the flexibility and quality of this approach for single/multi-layer materials by the fabrication and characterization of a graphene-based field effect transistor (Gr-FET) and a vertically-stacked heterostructure photodetector. The key concept of this technique is the utilization of a thin liquid film between a PDMS stamp and 2D monolayers as a dynamic bonding enhancer. The thin liquid film acts as an instant glue between the PDMS stamp and 2D materials, and subsequently peels them off from the substrates. After the thin liquid layer evaporates, the capillary force vanishes and leaves the 2D monolayers ready to be released onto new substrates. The transient adhesion-enhancing mechanism bypasses the
tradeoff between the peeling-off step and the releasing step, where the increase in adhesion fosters the peeling-off but hinders the releasing step\textsuperscript{19}. Since this method uses a distilled volatile liquid film instead of a solid sacrificial layer, both physical damage and chemical contamination are minimized in this process. In addition, the 2D monolayers can be released to a target area on the receiving substrate by aligning the PDMS stamp under an optical microscope, since they can be located optically or with the help of a marker. Using this method, we fabricated a Gr-FET by the direct transfer of an exfoliated graphene monolayer onto palladium-gold electrodes, which demonstrated a near-zero charge-neutral voltage and a high electron mobility of \(~48,820\) \text{cm}^2\text{V}^{-1}\text{s}^{-1} under room temperature, indicating an ultra-low surface doping and defect level incorporating from the transfer process. It is worth noting that this mobility level is the highest for graphene monolayers on SiO\textsubscript{2} surfaces, several times higher than those achieved by PMMA-based wet transfer with low-residue sacrificial layers, the previous best performers on SiO\textsubscript{2}\textsuperscript{20}. We also demonstrate the versatility of this quick and clean transfer technique in the fabrication of high-performance 2D heterostructure devices and have achieved fast optical response with a graphene/MoS\textsubscript{2}/graphene heterostructure photodetector. A capillary force model is discussed to reveal the role of liquid in this technique.

The capillary-force-assisted clean transfer process is illustrated in Figure 1. A thin PDMS film is first held over a beaker with boiling deionized (DI) water (or other solvents) for 3–5 seconds, to condense water on the PDMS surface. The PDMS is then quickly yet gently placed on the
substrate with 2D monolayers, which are prepared by the mechanical exfoliation method and confirmed by Raman spectroscopy. After forming a conformal contact with the SiO$_2$ substrate, the PDMS is quickly peeled off, starting from one side and maintaining a small angle between the PDMS and the substrate during the process. Firmly pulled by the capillary force, the 2D monolayers now adhere to the PDMS and are ready to be transferred onto the target substrate. Under an optical microscope, the PDMS stamp is aligned to the target substrate using a translation stage, so that the transferred 2D monolayers precisely overlap with the target region on the substrate. A simple pressing step will release the 2D monolayers. Since the soft PDMS is gently placed on the substrate, this releasing process will not damage the existing surface structures on the target substrate, such as microelectrodes and other 2D monolayers. By simply repeating this transfer process, 2D heterostructures can be built layer by layer.

To show the determining role of the capillary force in this new method, we compare the effectiveness of transferring MoS$_2$ flakes by dry and vapor-wet PDMS stamps in Figure 1b. The dry PDMS stamp can hardly pick up any MoS$_2$ flake from the substrate (Figure 1b, red path), whereas the vapor-wet PDMS stamp was able to transfer all flakes underneath to a clean SiO$_2$/Si substrate (Figure 1b, green path). It is also worth noting that this quick and easy process requires minimal instrumentation and time (Video S1 in Supporting Information). Moreover, in the case where the target material is sensitive to water, other volatile liquids can be used. (e.g. ethanol has been tried to transfer graphene in Figure S3, Supporting Information)
Figure 2 shows the optical microscope images and atomic force microscopy (AFM) characterization of the transferred results. Large-area graphene flakes containing both single and multilayer regions were transferred with high fidelity from a 300nm-SiO$_2$/Si substrate (Figure 2a) to another SiO$_2$/Si substrate (Figure 2b). The AFM image (Figure 2c) shows that both monolayer and multilayer regions of the graphene flake are wrinkle and crack free after the transfer. The low roughness of the flake surface indicates that it is clean of any particle or organic residues. Owing to the low adhesion energy on a dry PDMS, the graphene flakes can also be released to other types of substrate, such as a freshly peeled ultra-flat gold substrate (Figure 2i-k, surface roughness 0.6 nm), prepared by the fabrication method introduced elsewhere$^{21, 22}$. This transfer technique can be applied not only to hydrophilic materials like graphene but also to hydrophobic materials, such as molybdenum disulfide (MoS$_2$). Figure 2e and m show the optical microscope images of MoS$_2$ flakes transferred to a 300nm-SiO$_2$/Si substrate and a gold substrate with high fidelity, respectively.

A three-terminal back-gated graphene field-effect transistor (Gr-FET) was fabricated to characterize the electrical properties of graphene monolayers prepared by this method. The device layout is shown in Figure 3a. It was prepared by transferring a graphene monolayer directly onto pre-patterned electrodes (10nm/80nm/3nm Pd/Au/Cr, from top to bottom), which were defined by standard photolithography and metal deposition. This fabrication process avoids the direct contact of the graphene monolayer with any polymer solution, such as PMMA or
photoresist, and thus minimizes the contamination from polymer residuals. The quality of the transferred graphene was confirmed by Raman spectroscopy. As shown in Figure 3c, the spectrum features a symmetric Lorentzian-shaped G band centered at 1584.0 cm\(^{-1}\) and a 2D band at 2675.4 cm\(^{-1}\), with narrow peak widths at FWHMs (full width at half-maximum) of 11.6 cm\(^{-1}\) and 27.6 cm\(^{-1}\), respectively. The absence of the D’ band at 1620 cm\(^{-1}\) indicates a low density of random impurities or surface charges. This is also evidenced in the undetectable D band, which is caused by the disorders in sp\(^2\) hybridized carbon system. The average intensity ratio of the 2D/G is larger than 2, further confirming the high-quality of the monolayer graphene. No PMDS peak (~1410 cm\(^{-1}\)) shows up in the spectrum. All of these Raman characterization results are in good consistency with the high-quality monolayer graphene reported in the literature.

The cleanliness of the transferred graphene monolayer is also evidenced in the current-voltage characteristic of the Gr-FET device. Figure 3b shows the room-temperature conductivity as a function of the back-gate voltage (V\(_{BG}\)) of the device. The V\(_{BG}\) for the charge neutral point (CNP) is very close to zero (V\(_{BG,CNP}\) = 0.45 V with 300-nm SiO\(_2\) as gate oxide) in the as-prepared device, revealing a charge-neutral doping level (n\(_{CN}\)) of ~3 \times 10\(^{10}\) cm\(^{-2}\), where n\(_{CN}\) = \(\alpha V_{BG,CNP}\), with \(\alpha = 7.2 \times 10^{10}\) cm\(^{-2}\) V\(^{-1}\) derived from a parallel-plate capacitor model. The carrier mobility of the device can be extracted from a device model with the contact resistance and considered\(^{23}\). The mobility is found ~ 48,820 cm\(^2\) V\(^{-1}\) s\(^{-1}\) at room temperature. (Details in Supporting Information.)
Due to the simplicity of the transfer procedure, the exceptional cleanness of the sample surface, and the capability for repeated stacking of various 2D monolayers, this technique can also be used to prepare high-performance vertically stacked vdW heterostructures. Heterostructure devices have been demonstrated for several applications, including graphene stacked on h-BN for high-mobility FETs\textsuperscript{24}, tunneling field effect transistors (TFETs) and photodetectors\textsuperscript{25, 26}. Previous demonstrations of heterostructure applications mainly rely on PMMA-assisted transfer\textsuperscript{24} where the complete removal of polymer residues is challenging, or h-BN-assisted hot-pickup technique where the exposure of 2D materials to electrodes or environments is difficult. The capillary-force-assisted transfer demonstrated here is a quick and convenient method to build them.

As a proof-of-concept, graphene/MoS\textsubscript{2}/graphene (Gr/MoS\textsubscript{2}/Gr) vdW heterostructure devices (Figure 4a) were fabricated and characterized. The active regions were prepared by the successive transfers of a single-layer graphene film (yellow outlined area in Figure 4b), a 6nm-thick MoS\textsubscript{2} flake (green color region), and another single-layer graphene film (blue outlined area) from their original substrates onto pre-patterned electrodes. The positions of the transferred 2D flakes are carefully adjusted through a translation stage during each film transfer, to ensure a large Gr/MoS\textsubscript{2}/Gr sandwich area (the overlap of the blue and yellow outlined areas). In this region, the photon-generated electron-hole pairs in the MoS\textsubscript{2} layer can be separated and collected by the top and the bottom graphene electrodes (Gr\textsubscript{T} and Gr\textsubscript{B}) according to two driving
mechanisms. One is the force from the external electric field applied between \( \text{Gr}_T \) and \( \text{Gr}_B \), and the other is from the built-in electric field created by the asymmetric band structures. **Figure 4c** shows the photocurrent as a function of time measured under the alternating 532 nm-laser illumination (spot size \( \sim 1 \mu m \)) at four different optical powers (45, 90, 160 and 225\( \mu \)W). The photocurrents have fast temporal responses at both the rising edges (from OFF to ON) and the falling edges (from ON to OFF), and are stable at the “ON” states over long-term (~60 s) measurements. Such clear switching steps and the stable ON current indicate the absence of trapped states at the interfaces, which usually leads to a gradual change of photocurrent due to the slow charge transfer process. The fast photoresponse leads to the high operation speed as a photodetector. To characterize its high-speed performance (**Figure 4d**), we used a mechanical chopper to modulate the laser beam from 1k to 10kHz and used a lock-in amplifier (Stanford Research Systems, SR830) to analyze the short-current photoresponse signals. We observed no drop in photoresponsivity within our experimental temporal resolution (up to 10 kHz). Both \( V_{DS} \) and \( V_{BG} \) were set to zero during the measurement.

To quantitatively investigate the capillary-force-assisted transfer method, we developed a transfer model basing on adhesion force analysis. In general, the adhesion force between two surfaces contains three types of force – van der Waals force, electrostatic force, and capillary force. The former two play critical roles in dry conditions but are dramatically weakened in a liquid (humid) environment, due to the charge screening effect from the highly polarized water...
molecules\textsuperscript{27}. To calculate the capillary force, we consider the model of a capillary meniscus at
the touching region between a half PDMS cylinder and a flat surface, and a liquid bridge formed
from the water sandwiched between the PDMS and substrate (\textbf{Figure 5a,b}). The capillary force
is the result of both the surface tension around the circumference on the solid-liquid-gas interface
and the Laplace pressure inside the liquid bridge. If the bending radius of PDMS (\(R\)) is much
larger than the segment lengths of the liquid bridge (\(s_1\) and \(s_2\)), the capillary adhesion force can
be simplified as

\[
F_{\text{cap}} = \frac{2\gamma_L (\cos \theta_1 + \cos \theta_2)}{\alpha}
\]  

(1)

where \(\gamma_L\) is the surface tension coefficient of water-air interface (~72.9 mN/m at 20°C), \(\theta_1\)
and \(\theta_2\) refer to the water contact angles on the PDMS film and the substrate, and \(\alpha\) is the
angle between the substrate and the outer tangent line of the PDMS at the meniscus edge. The
detailed derivation of Equation (1) is described in the Supporting Information. It is significant to
note that the contact angle should be the dynamic dewetting contact angle (receding angle)
instead of the static contact angle in this equation, since the capillary bridge is sliding on both the
PDMS surface and the substrate during the peeling-off process. In general, the receding angle is
much smaller than the static contact angle, which leads to a positive (attractive) capillary force in
Equation (1) even for hydrophobic substrates such as pristine PDMS (with a static contact angle
of ~110°). To evaluate the receding angles on different materials, we performed a tilted-plate
measurement by using a digital goniometer integrated with a syringe needle to record the droplet of deionized water placed on the surfaces of tilted substrates. Water drops on PDMS, glass and highly oriented pyrolytic graphite (HOPG) substrates give receding angles of 50°, 23°, and 49°, respectively (Figure S5 in Supporting Information). Here, HOPG is used to mimic the graphene flake surface. We also measured the angle α by measuring the contact angle between a thin PDMS film (~2mm in thickness) and the SiO₂ substrate with different peeling speeds, and found it in the range of 5°~30°. Using Equation (1), we can estimate the capillary forces between PDMS-water-glass and PDMS-water-HOPG interfaces to be in the range of 0.45~2.6 N/m and 0.38~2.2 N/m, respectively.

To verify this capillary force model, we measured the adhesion force between a thin PDMS film and various substrates under dry and wet conditions. A laboratory-developed peeling-off apparatus is used to record the peeling-off force (F Peel) needed to separate a PDMS strip from different substrates at velocity v. As shown in Figure 5b, a typical specimen contains SiO₂ regions and a 4 mm-wide HOPG region, which is formed of thin HOPG flakes flattened on the SiO₂ substrate. Before measuring the dry-condition F Peel, the freshly prepared PDMS strips were placed on the substrate directly, followed by 1-hour of relaxation to release stress. To measure the wet-condition F Peel, the PDMS strip was hung over a beaker with boiling water for a few seconds to condense water, and then placed on the sample substrate for the immediate measurement. F Peel is measured at a low peeling-off rate of 100μm/s. Both the mechanical energy
stored in the PDMS film and the influence from the weight change in the vertical part of the PDMS strip were compensated in the data analysis. The black curve in Figure 5c shows the $F_{\text{peel}}$ at the dry condition. It starts at around 550 mN/m on SiO$_2$ and drops steeply to ~40 mN/m when the separation region reaches the SiO$_2$-to-HOPG boundary. At the HOPG-to-SiO$_2$ boundary, $F_{\text{peel}}$ gradually returns to 550 mN/m. Then the separation front starts to move on again and $F_{\text{peel}}$ remains at around 550 mN/m. Since the $F_{\text{peel}}$ on glass stays at the similar level throughout the operation of the experiment (~3 min), we believe that the swelling effect in PDMS, which is at the time scale of hours, can be ignored here. The adhesion energy between HOPG and PDMS (~40 mJ/m$^2$) is much lower than that between a graphene monolayer and SiO$_2$ (~240 mJ/m$^2$), which explains why PDMS cannot pick up a graphene monolayer directly from a SiO$_2$ substrate.

In the wet-condition measurement, $F_{\text{peel}}$ is lowered to ~450 mN/m on the SiO$_2$ surface, but on HOPG surface it significantly increases to ~200 mN/m. It is large enough to overcome the binding energy between graphene and SiO$_2$, leading to the peeling-off of graphene from the substrate. Moreover, the $F_{\text{peel}}$ in our experiment is limited by the large surface roughness of HOPG flakes (more details in Supporting Information), which increases the average $a$ and lowers $F_{\text{peel}}$. The adhesion energy on a flat graphene surface can be larger than 200 mJ/m$^2$, which may be utilized to transfer 2D materials from more adhesive substrates, such as metals. We tested this transfer method on graphene monolayers and MoS$_2$ monolayer domains prepared by chemical vapor deposition (CVD) methods on copper films and SiO$_2$ substrates, respectively.
Since the adhesion energy between graphene and Cu surface is high (~720 mJ/m$^2$), only about half of the total area was transferred. (Figure S7 in Supporting Information). In the CVD-MoS$_2$ case, all MoS$_2$ domains were transferred (Figure S8).

In summary, a simple and clean stamp transfer method for 2D materials is developed. Using a thin layer of water as an instant yet temporary glue, the adhesion energy between PDMS and 2D materials can be significantly increased during the peeling off process and restored shortly to facilitate the releasing process. The Gr-FET device characterization reveals a very low impurity level on graphene monolayer after the transfer. High-quality Gr/MoS$_2$/Gr vertical heterostructures with clean interfaces are fabricated to show the convenience of this method for constructing complicated heterostructures. Moreover, the mechanism model study discloses that higher capillary force can be achieved by using smaller contact angle $\alpha$ between PDMS and the substrate, which will stimulate further investigation of using this method for transferring large-scale 2D materials, and accelerate the development of 2D material studies.
Method:

**PDMS preparation.** A standard PDMS preparation recipe is used. In a clean plastic tube, mix PDMS and hardener at a ratio of 9:1 (Dow Corning, Sylgard 184). Using the non-cloth end of a plastic swab to mix the solution for 5 mins. Clean glass slides with piranha solution (98% H₂SO₄ : 30% H₂O₂ = 3:1) for 1 hr, rinse with DI water for 2 min. Place the cleaned slides in a petri dish and pour the mixed PDMS solution on the slides till ~2 mm in thickness. Wait for 1 hr to vent bubbles, then bake at 50°C for 10 hrs. Cut PDMS into desired sizes and clean in isopropyl alcohol (IPA) and DI water sonication for 2 min each before using.

**FET electrode preparation.** The Si wafer (with 300 nm thermal oxide) is first cleaned with piranha solution for 1 hr, then rinsed with acetone, IPA, and DI water, respectively. Standard photolithography and e-beam evaporation are used to define the electrodes (10 nm/80 nm/3 nm Pd/Au/Cr, from top to bottom). After lift-off in acetone, the substrates are cleaned in IPA and DI water sonication for 10 min each before using.

**2D material transfer.** The graphene and MoS₂ flakes are prepared using the standard mechanical exfoliation method on 300 nm SiO₂/Si substrates. ~50 mL DI water in a 100 mL beaker is preheated on a hot-plate (Fisher Scientific, Isotemp) at 120°C to generate stable steam. The cleaned PDMS piece is held over the steam for 3–5 sec to condense water, then placed gently on the SiO₂/Si substrate. Note that long-time water condensing may lead to distorted transfer results. Wait for a few seconds to relax the film and peel it off from one side. The 2D flakes are attached to PDMS and are ready to be released. The release is conducted under an optical microscope to locate the position of electrodes. A simple stamping procedure is sufficient to leave the 2D flakes to the substrate.

Acknowledgments:

This work is supported by NSF Grants CAREER DMR-1654746 and DMR-1649795. We thank Dr. Nathaniel Gabor for the use of optical microscope alignment setup.

Supporting Information:

Additional information on comparison with the conventional transfer method, capillary force model and derivation, transfer results with other liquids, influence from liquid layer thickness to the transfer results, dynamic contact angle measurement, angle α measurement, transfer of CVD
graphene and MoS$_2$ results, HOPG flake characterization, FET analysis, role of water in the transfer method, influence of flake thickness, and two supporting videos.
References:


Figure 1a, Schematic diagram of the capillary-force-assisted transfer procedure. b, The comparison between the adhesion capability of a dry polydimethylsiloxane (PDMS) film and a wet PDMS film. The dry one cannot peel off any molybdenum disulfide (MoS$_2$) flakes from the SiO$_2$ substrate (upper row, red path), while a wet PDMS film can pick up all flakes (lower row, green path). The picked-up flakes can be transferred to the target substrate (right). All scale bars are 1cm. A standard PMMA-assisted method can be found in Supporting Information for comparison.
Figure 2. Optical microscope images and atomic-force microscope (AFM) cross-section images of graphene and MoS$_2$ flakes transferred from the preparation substrates (300nm-SiO$_2$/Si, labeled as SiO$_2$) to the target substrates (300nm-SiO$_2$/Si and super-flat gold substrates). All scale bars are 10 μm.
Figure 3. Electrical and Raman characteristic of a graphene FET prepared by direct transferring of a graphene monolayer onto electrodes. a, the optical microscope image of the device layout. The electrode contains 10nm Pd, 80nm Au, and 3nm Cr. b, source-drain current of the Gr-FET vs the back-gate bias $V_{BG}$, measured under ambient conditions at room temperature. The thickness of the SiO$_2$ gate oxide is 300nm. $V_{DS}$ is 10mV. The charge-neutral-point gate voltage is 0.45V. c, Raman spectrum of the graphene channel region. d, The total resistance $R_{tot}$ vs $V_{BG}$ with both experiment data (squares) and modeling result (line).
Figure 4. Photocurrent generation in a vertically-stacked graphene-MoS$_2$-graphene heterostructure. a, the schematic illustration of the heterostructure device layout. b, an optical microscope image of the device, prepared by successive transferring of a monolayer graphene bottom electrode (yellow-outlined region), a few-layer MoS$_2$ flake, and a graphene top electrode (blue-outlined region) onto the electrode. c, the photocurrent measurement of the sandwiched region (intersection of the blue- and yellow-outlined regions), excited by a 532-nm laser with various powers. The characterizations were performed under ambient conditions, with both the bias voltage ($V_{DS}$) and the back-gate voltage ($V_{BG}$) set zero. d, the frequency-dependent photocurrent measurement.
Figure 5. Capillary force model analysis and experimental measurement. a, schematic of the adhesion force measurement using a thin PDMS stipe stamp on the target substrate, which contains a 4 mm-wide highly-oriented pyrolytic graphite (HOPG) region. b, mechanism schematic of capillary interaction between PDMS and substrate. c, adhesion force measurement under dry and wet conditions, measured at a peeling-off rate of 100μm/s. The adhesion force on HOPG is dramatically increased under the presence of a meniscus bridge.
Graphic for manuscript

75x39mm (300 x 300 DPI)